nlgispokesman

Scientific Detection In Grease Lubrication Problems
By R. J. RONAN and M. C. McCLAREN

A Rapid Method of Analysis and Evaluation of Lubricating Grease By F. E. CHAMBERLIN, C. F. CARTER and J. L. DREHER

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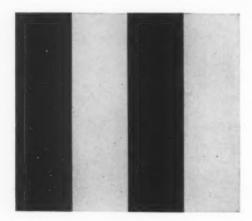
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News About NLGI

NLGI Begins Second Survey, on 1958 Production of Lubricating Greases and Fluid Gear Lubricants

The second production survey is about to be taken. Officially known as the NLGI Survey on Production of Lubricating Greases and Fluid Gear Lubricants for the Calendar Year of 1958, questionnaires will be mailed to Active (manufacturing) members in mid-January for another voluntary compilation of data by and for the industry.

Production in pounds for the twelve months is the basis of the survey . . . this system is to eliminate the possibility of duplication that sale and re-sale might bring if marketing figures were totalled. Last year's gathering on 1957 production showed that the industry produced over one billion pounds of lubricants. An excellent showing of three-fourths of the membership replied on the first survey and an estimated 85 per cent of production facilities were represented.

Newest development this year will be the inclusion of all NLGI Canadian member firms, who will participate simultaneously with U. S. firms. Canadian totals will be published at the same time American statistics are given, but in a separate column.

Part of the success of the first survey and its fine member response is attributed to the care with which the mechanics of this new service were inaugurated—over a year was spent in careful consideration of every step, and in the selection of an agency to gather the data. Based on an excellent record of accuracy and reliability rendered other associations for similar surveys, the management services division of the nation-wide accounting firm of Ernst and Ernst was selected. E. & E. has again been chosen to handle the 1958 survey. A review of the procedures:

No one connected with the Institute has any contact with the survey, the gathering or totaling the statistics.

All forms mailed to members go by registered mail—they are returned to E. & E. by registered mail. Forms are destroyed after totals are run. All forms are on E. & E. stationery and are anonymous—the one exception this year will be Canadian members. They will have the word "Canada" typed in the upper left margin.

After the closing on March 31, the management services division will compile the figures and then offer NLGI's national office the grand totals. These figures will be mailed immediately to all member firms, in every classification.

With a second set of figures to compare the survey will begin to offer historical significance. There is considerable feeling too, that the 1958 figures will render a more complete showing of the industry as traditionally, surveys of this nature tend to gain participation with each succeeding year.

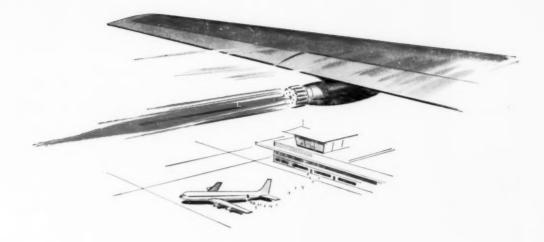
Scheduling of the survey includes mailing of forms to members in mid-January, closing on March 31, and distribution of data in late April.

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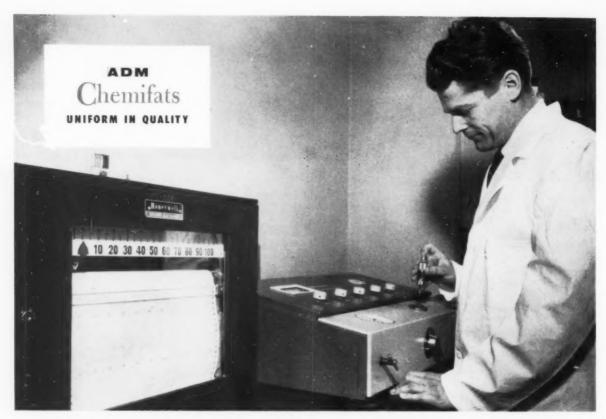
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THE COVER

The value of the infra-red spectrophotometer, the machine shown on the cover. is discussed in the article "Scientific Detection in Grease Lubrication Problems" by Ronan and McClaren, beginning on page 460. The machine's operation is based on the following principles-organic liquids will absorb light of various wave lengths. The amount of absorption of light of a specific wave length is a unique property of the particular organic liquid involved. By passing light of varying wave lengths through a liquid and measuring the amount of absorption at each wave length, a chart is obtained which is as specific to that liquid as finger prints are to people.

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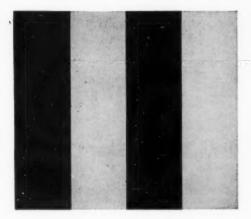
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NLGI PRESIDENT'S PAGE

By F. E. ROSENSTIEHL, President



Chassis Grease-A Declining Market?

Those of us who heard the presentation made by Mr. W. M. Drout, Jr., at the recent NLGI annual meeting on "Market Potential for Automotive Grease" should be alarmed at the gloomy outlook presented. Mr. Drout's paper reflected a 56 per cent reduction in the number of chassis grease fittings in the past seven years. Further, the automotive manufacturers feel rather strongly that complete elimination of fittings is only a matter of time, predictions ranging from three to five years as to when this will be accomplished.

Certainly these figures paint a dark picture for the future of automotive greases, even taking into account the number of older cars, with their many grease fittings, still on the road. Unfortunately, recent advances in the field of permanently-lubricated or essentially friction-free bearings, make this development seem all-too-possible.

Another factor that has decreased automotive grease consumption is the longer time interval car owners allow to clapse between greasings. Mr. Drout points out that the majority of motorists (from 70 to 90 per cent) generally grease their cars at the same time they change the oil. Based on past performance, this increasing grease interval may be expected to continue upward to approximately 2,700 miles in 1965, as compared to 2,200 miles in 1957.

On the brighter side is the fact that motor vehicle registration is expected to continue its steady climb, increasing the potential automotive grease market 30 per cent by 1965. Also, the annual miles driven per vehicle, which directly affects grease consumption, should remain essentially constant through 1965.

Yet, despite the rising number of cars on the road, the inevitable reduction in grease fittings and extension of greasing intervals will seriously undercut the future market for automotive grease, particularly in the passenger car field. (It is not anticipated light and heavy trucks and buses will be an important factor in this declining market.)

As nearly as can now be estimated, this market will decrease from 120,000,000 pounds per year to approximately 39,000,000 pounds in 1965, a decrease of 67 per cent.

Just what we in the grease industry can do to prevent this decline is problematical. Already the manufacturers of self-lubricated bearings are predicting "cars that don't have to be greased in 1960," and rumor has it that a car manufacturer will, indeed, introduce an automobile without any chassis lubrication fittings in 1960.

The problem is a serious one, and I do not raise it here to offer some ready solution. But, if we are to find a solution, if we hope to open new markets that will help offset this loss, then we will certainly not do so by ignoring the harsh facts that lie almost immediately ahead.

Scientific Detection In Grease Lubrication Problems

By: R. J. Ronan and M. C. Mc Laren
The Texas Company

Presented at the NLGI 26th annual meeting in Chicago, October 1958

Abstract

The writers discuss the development of the "technical service man" for grease lubrication and show how this development has assisted machinery designers and lubricating grease users. A number of modern analytical devices are described briefly and their use in the solution of practical problems is discussed.

HE STATUS OF the art and technology of lubricating greases has changed radically in the last generation and greases have been improved greatly to meet the increasing demands placed upon them. Improvements in lubricating greases are a tribute to the work of the grease researchers, the bearing and machinery designers, and the lubrication engineers of grease users, including Government agencies; but there is another group which has had its share in the development of the art. In the smaller grease companies, the researcher leaves his laboratory to make his own field observations, and returns to the laboratory to develop modified or improved greases based on his

observations. In the larger companies, this field work has been taken over by specialists who are usually titled "Technical Service Men."

The technical service men act as the eyes of research in determining, in the field, what characteristics of the lubricant require improvement. After a lubricant is developed or modified, it is tested as exhaustively as possible under laboratory conditions. The technical service man then takes the newly developed product into the field and determines under actual service conditions whether it performs as intended, and what its service limitations are.

The technical service man acquaints the designers of bearings and machinery and the lubrication engineers of grease users with the new developments in grease technology so that advantage can be taken of them in service. On the other hand, the technical service man finds out from the designers and users where presently available products are deficient, for the guidance of the grease researchers.

In his day-to-day work, the technical service man has at his command the facilities of a modern petroleum laboratory. In such a laboratory task forces of chemists, engineers, metallurgists and grease technologists work together to determine the reasons for service difficulties and to recommend steps which can be taken to avoid them. Generally, both short-term "fixes" and long-term changes in lubricants and service environment are recommended, since Industry demands a practical, immediate answer, as well as a long-range solution to such problems.

The modern laboratory has at its command a wide array of analytical instruments which can shed light on problems which were completely beyond the scope of the analyst of a former generation. Specific examples of the solution of lubricating problems using modern analytical tools will be given in this paper to indicate the breadth of technical service activities. It should be kept in mind, however, that all of the familiar physical and chemical grease tests are still in constant use and are still helpful to the analyst.

Infra-Red Absorption Spectroscopy

Organic liquids will absorb light of various wave lengths. The amount of absorption of light of a specific wave length is a unique property of the particular organic liquid involved. By passing light of varying wave lengths through a liquid and measuring the amount of absorption at each wave length, a chart is obtained which is as specific to that liquid as finger prints are to people. Figure 1 (as shown on cover) is a view of an infra-red spectrophotometer.

In one problem, deposits were detected in a machine which used both a soluble oil and a grease. The labora-

tory was asked whether the grease or the soluble oil was responsible for the deposit formation. The petroleum laboratory fixed the responsibility by separately extracting the total fatty acids from the deposits, the grease and the soluble oil, and subjecting them to infrared absorption spectroscopy. As may be seen from Figure 2, the instrumental traces from the total fatty acids of the deposit and the grease are quite similar, while that of the soluble oil is very different. The laboratory concluded therefore that the grease was responsible in some manner for the deposit formation.

Emission Spectrography

Emission spectroscopic analysis is based on the fact that atoms of most elements when brought to an incandescent state emit radiation of various wave lengths which is characteristic of the element. An electric arc or spark is generally used to create the incandescence. This radiation is then sorted by an instrument called a spectrograph, pictured in Figure 3, which uses a prism or diffraction grating as the actual wave length "sorter." A photographic plate or photo electric cell detects the radiation of various wave lengths entering the spectrograph through a narrow slit. Since metallic elements can be detected in a sample when they are present in as small an amount as a few parts per million, the emission spectrograph has become a work horse in the modern laboratory for the detection of wear metals and contaminating elements. The emission spectrograph gives answers in a matter of minutes which could only be obtained formerly by days or weeks of tedious and complicated analysis.

X-Ray Diffraction

Another analytical tool makes use of X-Rays as the

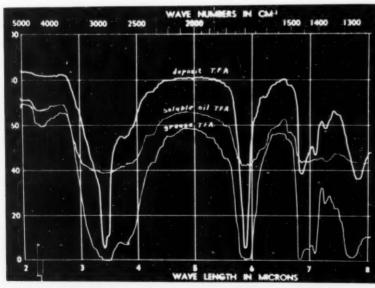


FIGURE 2 Infrared absorption spectrographs of three fatty acid samples

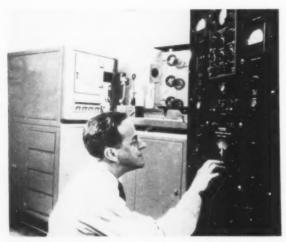


FIGURE 3, emission spectrograph

source of radiant energy. Since every crystal diffracts X-Rays of known wave lengths in a different manner, the X-Rays can be used to determine the nature of a crystalline material. This is a most important characteristic of this analytical method since conventional methods can only tell if elements are present and not how they are combined. A view of the X-Ray film diffraction equipment is shown in Figure 4.

In a particular case a customer submitted the roller bearing portrayed in Figure 5. The original bearing and its lubricant were liberally contaminated with both red and black material. The problem was to determine the nature of the contaminant and its origin. Using the X-Ray diffraction technique, the laboratory analyst determined that the contaminant was an ironoxygen compound and specifically alpha ferric oxide, which is characteristic of fretting corrosion caused



FIGURE 5, corroded tapered roller bearing



FIGURE 4, X-ray film diffraction equipment with electronic circuit panel

by heavy reversible loading or vibration with little movement. Had ordinary rusting occurred, the contaminant would have been hydrated ferric oxide and the technical service man would have sought a source of water ingress to the bearing.

In the absence of the X-Ray diffraction technique, the analyst would only have been able to report that an iron oxide was present, but he would not have been able to differentiate between the possible ferric oxides and thus pinpoint the source of the difficulty.

Spot Tests for Grease Identification

As illustrated in Figure 6, unbelievably small precision ball bearings are now made for instruments and control mechanisms. Although the quantity of grease required is microscopic, the quality of the grease for a particular application is extremely important. Since different greases were used in a certain bearing plant,

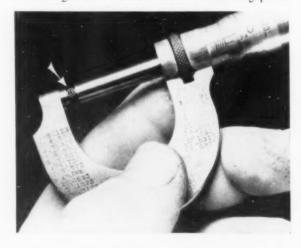


FIGURE 6, instrument ball bearing 0.10 inch OD



FIGURE 7, spot test for grease identification



FIGURE 8, micro hardness tester

JANUARY, 1959

confusion occurred and the manufacturer's problem was to avoid confusion by being able to identify the grease in random sample bearings from his inventory.

The petroleum laboratory solved the manufacturer's problem by developing a simple spot test. The bearing, with its grease charge intact is placed in a tiny beaker covered with a paper strip impregnated with a suitable chemical reagent. When warmed for about two hours, the strip develops a distinctive color that can be matched with a similar strip from known greases. (Figure 7)

The Tukon Hardness Tester

Since most common engineering materials are mixtures or combinations of several compounds, the hardness of the bulk material will be of some value intermediate between the hardnesses of the several compounds present. The ordinary hardness measurement is only an average determination over a relatively large area. It is neither confined nor precise enough to measure the hardness of tiny material components. Fortunately for the laboratory technologist, the very special hardness measuring device illustrated in Figure 8, has become available in recent years. Figure 9, shows hardness tests taken in a cross-section across the eye of a small sewing needle. Figure 10, is a photomicrograph of tin base babbitt showing that the square white tin-

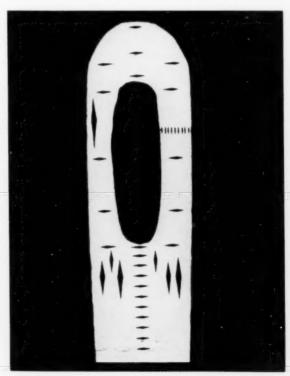


FIGURE 9, micro hardness of needle eye



FIGURE 10, photomicrograph of tin base babbitt

antimony crystals are much harder than the soft tin matrix in which they are imbedded.

A manufacturer complained of the short useful life, afforded by a particular spur gear, the usual inference being that its high wear was caused by an inadequate lubricant. Figure 11, presents a cross section of a rep-

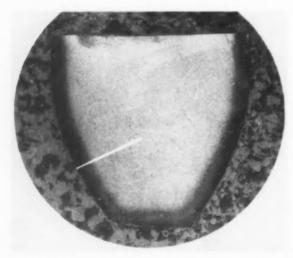


FIGURE 11, photomicrograph of section of spur gear tooth

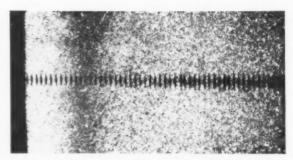


FIGURE 12, hardness measurements near wearing surface of spur gear tooth

resentative gear tooth which the laboratory molded into a mottled bakelite holder, polished to a flat surface and etched to reveal grain structure. What appears to be a simple white line is actually composed of more than 50 closely-spaced and diamond-shaped hardness impressions. Figure 12, presents a magnified section of the white line. To the unaided eye, the lengths of the impressions seem to increase rather uniformly from

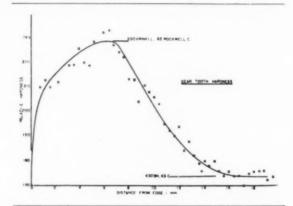


FIGURE 13, variation in gear tooth hardness

the tooth wearing surface. From Figure 13, however, which presents a plot of the accurately measured hardness, it will be seen that the tooth wearing surface is actually a relatively soft skin, with the desired hardness buried more than a half millimeter within the tooth where it is of little use. Further investigation in the manufacturer's plant showed that the atmosphere in a heat treating furnace was oxidizing and decarburizing the tooth surface. Correction to a neutral atmosphere restored the surface hardness of the gears and eliminated the wear problem.

Chromatography

An old tool of the analyst, the chromatographic column, has been greatly expanded in usefulness in the last few years. When mixtures of organic liquids are passed through a column packed with an absorbent



FIGURE 14, electron microscope

medium such as alumina, silica gel or charcoal, the components tend to absorb at different rates and can be washed out with different solvents. Thus, separations can be made with relative ease of materials which heretofore stubbornly defied splitting apart. With this technique, additives or oil-soluble contaminants can be removed from the oily component of greases. An ASTM sub-committee is also considering absorption chromatography for the separation and analyses of lubricating grease itself.

Electron Microscope

The electron microscope (Figure 14), has pushed back the horizons to which the laboratory worker can see. Changes in grease structure with varying degrees of service can now be observed (Figure 15). In addition, models are available with attachments by which single crystalline particles can be isolated and identified by their diffraction patterns. This technique should find many uses in the identification of finely-divided traces of contaminants and wear debris.





FIGURE 15, electron micrographs of unused grease at left and used grease at right

Service Tests

In addition to the use of special laboratory analytical instruments, the petroleum laboratory makes all kinds of special set-ups to simulate service conditions with the object in mind of finding out how lubricants perform in service. Where available products cannot satisfy the requirement of these service tests, new products are developed and screened by them. Humidity cabinet tests, vibrating wheel bearing tests, pumpability tests and track-roll mud bath tests are a few examples of the wide variety of such tests that have been developed.

Summary

The writer discusses the development of the "technical service man" for grease lubrication and shows how this development has assisted machinery designers and lubricating grease users. A number of modern analytical devices are described briefly and their use in the solution of practical problems is discussed.



R. J. Ronan attended St. Patrick's Academy, St. Francis Prep., Pratt Institute and in 1939 received his BS in chemistry from Polytechnic Institute of Brooklyn. From 1930 to 1933 he was employed by the Texas Co. as a messenger-clerk. From 1933 to 1939 he worked as a dye-stuff chemist for Dye Specialties. Since 1939 Mr. Ronan has been employed by the Texas Co. He was an ans-

lytical chemist from 1939 to 1940 in Beacon, N. Y. In 1940 he went to the standardization department in Beacon where he stayed until 1942. He transferred to New York in 1942 and worked in technical services until 1952. From 1952 to 1956 he was supervisor in the field services department. He is now regional manager of technical services. Mr. Ronan is a member of Phi Lambda Epsilon

About the Authors

M. C. McLaren attended Ashtabula Harbor school and in 1928 obtained his AB degree with a major in chemistry from Onerlin college. From 1928 to 1943 he was employed by the lubricating oil research and

processing groups of the Texas company at Port Arthur. Since 1943 he has been working in the products application and field service departments of the Texas research center at Beacon, New York.





APPARATUS used to extract oil from greases for viscosity measurements

A Rapid
Method
of
Analysis
and
Evaluation
of
Lubricating
Greases

By: F. E. Chamberlin

C. F. Carter

J. L. Dreher

California

Research Corp.

REQUENTLY IT IS necessary to perform grease analyses in order to evaluate competitive greases, to confirm the composition of our own products, and to assist in field problems. With some minor changes, ASTM Method D 128 has been the principal method for analyzing lubricating greases since 1922, but the procedure has these disadvantages: (1) the services of an experienced grease analyst are required, (2) the procedure is time consuming and expensive, (3) under certain conditions some parts of the procedure may give incomplete separations, e.g., the base oil component, and (4) the procedure is not applicable to newer types of thickeners.

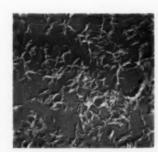
Although it is not the intention of the authors to propose a method to replace ASTM D 128, much can

be learned about the composition and physical properties of a lubricating grease from a few well-chosen tests which can be made in a short time. In addition to financial savings due to fewer man-hours per analysis, the time saved is especially important when giving laboratory assistance to field problems. It is well known that the quicker that information can be obtained in the laboratory and passed along to the field representative and to the customer, the more valuable that information becomes.

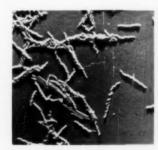
Considerable attention has been given to selection of tests which require only a small amount of sample. A one-pound sample has been found adequate for test work, and this size sample is usually convenient for the field representative to obtain.



Lithium Hydroxystearate



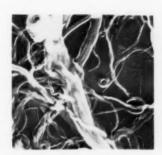
Sodium N-Octadecyl-Terephthalamate



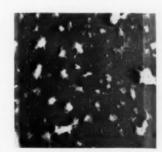
Calicium Tallowate



Lithium Stearate



Sodium Tallowate



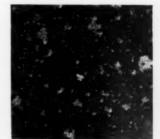
Aluminum Stearate



Bentonite Clay



Sodium Stearate



Silica Gel

FIGURE 1, typical photomicrographs of various types of thickeners X 7500

Normally, test data are obtained on the oil, thickener, and grease properties as follows:

Oil Properties

Test Method

Viscosity

Sufficient oil (10-20 ml) for kinematic viscosities at 100°F and 210°F is obtained by vacuum filtration. A sintered glass funnel or a Buchner funnel is satisfactory for this purpose. The oil can be collected in a small vial inside the vacuum flask in order to conserve the amount of grease required. The oil extraction can be accelerated by use of heat.

Data and Interpretation

1. Base oil viscosity:

2. Viscosity Index

Some lubricating grease additives are soluble in the base oil and will also be extracted in the filtration procedure. The viscosity of the oil obtained will then be the actual viscosity of the liquid component of the grease. This is not always the case when the oil is obtained by the ASTM procedure.

After some experience with the vacuum filtration, the operator is able to obtain some indication of the bleeding tendency of a grease by the rate at which the oil is extracted.

Thickener Properties

Test Method

Spectographic Analysis

A method specific for greases, comparable to the proce-

Data and Interpretation

This analysis indicates the metallic component of soaps or other thickeners. The presdure given by Key and Hoggan,³ is used. Semi-quantative values can be obtained.

Sulfated Ash

ASTM D 128-47 Section 6. The use of this test, togeth-

or with the spectrographic analysis, eliminates the lengthy wet analysis for metals.

Dropping Point ASTM Method D 566-42.

ence of fillers, such as zinc oxide, aluminum oxide, lead soaps, and molybdenum disulfide can be determined.

The total metallic content of a lubricating grease is determined, quantitatively by this test. If there are no fillers present, a good approximation of the thickener content can be calculated from the sulfated ash value.

As indicated by the following table, the dropping point can identify the type of thickener or, at least, eliminate the possibility of some types of thickeners.

	Dropping Point
Type Thickener	$Range, {}^{\circ}F$
Inorganic and syn- thetic organic	500+
Sodium soap	350-450
Barium soap	330-450
Lithium soap	330-390
Calcium 12 hydroxy stearate	270-300



About the Authors

F. E. CHAMBERLIN joined California Research corporation in 1956 and is a member of the grease research and development group. Previously he was with Sinclair research laboratories at Harvey, Illinois. Mr.

Chamberlin obtained his BA degree in chemistry from the University of Kansas City in 1949. He is a member of the Northern California section of American Society of Lubricating Engineers.

C. F. Carter has been engaged for the past sixteen years in the development and manufacture of lubricating greases with the California Research corporation. Mr. Carter obtained his BA degree from Polytechnic college of engineering in 1934. In 1935 he

was employed by California Research, operating company of the Standard Oil company of California. Mr. Carter has been a keenly interested member of ASLE, and is also a frequent contributor to the journal of the National Lubricating Grease Institute.





About the Author

J. L. Dreher is group supervisor of the grease research and development laboratory of California Research corporation. He joined the company in 1945. Dreher obtained his AB degree in chemistry from the University of California in 1935 and began working for General Petroleum corporation.

In 1943 he joined the Metallurgical Laboratories (Manhattan Project), Chicago University, and was then employed by Hanford engineering works (a subsidiary of E. I. Du Pont de Nemours and Co.) in 1944. He is a member of ACS, ASLE, and American Association for the Advancement of Science.

Aluminum soap Calcium soap (hydrated) 180-240

When sufficient interest in a grease warrants specific identification of the type thickener, electron microscope photomicrographs can be used to establish the structure of the fibers. By comparison with a catalog of known fiber structures, as shown in Figure 1, the type of thickener can

Data and Interpretation
In addition to establishing

the NLGI grade, the spread

between the unworked and

worked penetrations can be

usually be determined.

Electron Microscope

Although this apparatus is used principally for research purposes, it can be used for analytical work. Any of the normal techniques are satisfactory for obtaining electron microscope photomicrographs.

Grease Properties

Test Method

Unworked and Worked Penetration

ASTM Method D 217-52T ASTM Method D 1403-56T (One-Quarter Scale)

ASTM Worker

100,000 Strokes Federal Test Method Standard No. 791, Method 313.1 The primary purpose of this test is to measure mechanical stability. However, it also gives a broad indication of the type of thickener used. Greases prepared with 12 hydroxy-stearate soaps, inorganic thickeners, and synthetic organic materials usually have good mechanical stability. Most greases made from fatty materials, such as stearic acid and tallow, etc., have poorer mechanical stability.

Test Method

Behavior Over a Wide Temperature Range

In this test a sample of grease is spread uniformly along a graduated heating bar in a thin layer.

Data and Interpretation

Observation of the grease when at equilibrium will give the following information:

- 1. The temperature range through which a grease retains its body gives a general idea of its maximum functional operating range.
- 2. The temperature of liquefaction can be observed to give a quick approximation of the dropping point.
- 3. The useful property of a grease to regel after liquefaction and still retain a grease structure can be established by this test.
- 4. The presence of water can be detected by a bubbling action at the hot end of the

Boiling Water Test

A small sample of grease is placed in boiling water and observed. This test provides a rapid method for determining the water resistance of a grease.



A RESEARCHER analyses a grease sample in emission spectrograph.

Test Method

Thin Film Test

The literature^{2, 5} gives descriptive methods for this test.

Centrifugal Filtration Test

The procedure for this test is described in an earlier issue of the NLGI Spokesman.¹

Data and Interpretation

The results of this test are indicative of the oxidation stability and bearing performance of the grease and the volatility of the base oil.

The advantages of this test are the small amount of sample and the short test time required to determine the bleeding tendency of a grease.

The value of this method lies in speed and economy. In most cases, it has been found that an approximation of the grease composition usually suffices. As a general practice, the time required to determine the exact amount of thickener is not warranted if an approximation within 10 per cent to 20 per cent of the actual value can be obtained quickly.

In practice, some combination of the preceding tests usually gives all the information necessary to establish the approximate composition and to evaluate an unknown grease. The selection of tests depends largely upon the degree of interest in a sample and any specific properties that are thought to warrant investigation.

The tests which have been found most useful are shown below, together with approximations of the number of man-hours required to perform the tests.

Operation	Man-Hours
Oil Separation and Viscosity Determination	0.6
Spectographic Analysis	1.0
Sulfated Ash	0.5
Dropping Point	0.8
Penetrations	
Unworked	
Worked 60 Strokes	0.3
Worked 100,000 Strokes	
Heating Bar Test	0.1
Total	3.3

The estimated man-hours required for ASTM Method D 128 are given below.

Operation	Man-Hour
Separation of free fatty acids, fatty acids f	from
soap, and fatty acids from fat	2.0
Identification of acids	2.0
Separation of petroleum and unsaponifiabl	C
matter	0.5
Viscosity determination	0.5
Sulfated ash	0.5
Identification of ash	0.5
To	tal 6.0

Comparison of the working time for the two methods shows a considerable savings for the proposed rapid method. The estimated time of six man-hours required for ASTM Method D 128 does not take into considera-



THE authors F. E. Chamberlin, J. L. Dreher and C. F. Carter are shown discussing laboratory data.

tion the time required to obtain such grease properties as consistency, dropping point, mechanical stability, and other tests which are necessary in order to evaluate grease performance. The rapid method provides this information as part of the procedure.

References

- Dreher, J. L., and McClellan, A. L., NLGI Spokesman, 10, No. 12, 3 (1956).
- 2. Dreher, J. L., NLGI Spokesman, 21, No. 2, 5 (1957).
- Key, C. W., and Hoggan, G. D., Analytical Chemistry, 26, No. 12, 12 (1954).
- McClellan, A. L., and Cortes, J., NLG1 Spokesman, 20, No. 6, 9 (1956).
- 5. Woods, H. A., NLGI Spokesman, 20, No. 12, 3 (1956.)

1958 PRODUCTION SURVEY FOR LUBRICATING GREASES AND FLUID GEAR LUBRICANTS

THE NATIONAL Lubricating Grease Institute will again sponsor a survey for the industry—to compile production data for the calendar year of 1958. Active (manufacturing) members will be mailed questionnaires in mid-January, 1959, and totals will be compiled in March. All handling and processing of survey questioners will be by the management services division of Ernst & Ernst, nationwide certified public accountants. No one affiliated with NLGI in any fashion will have contact with questionnaires and the forms will be destroyed after totals are made.

Last year, the Institute held its first survey for 1957 production and received a heartening 76 per cent return. This was considered excellent for a first survey and the Institute's board of directors feels that the percentage of returns will be even higher for the 1958 survey, making the data more valuable. Data will be made available to all classifications of membership in April.

Future Grease Requirements-2

Current and Future Military Grease Requirements

By R. J. Horwath
Military Petroleum Supply Agency

Presented at the NLGI 26th annual meeting in Chicago, October 1958

Abstract

This paper presents the statistical data for the various greases purchased by the Military Petroleum Supply agency for the military departments. Included is the overall volume and dollar value of greases versus the total volume and dollar value of all petroleum and related products procured during the fiscal years of 1957 and 1958 as well as data relative to small business participation in these procurement programs. Detailed for these two years are the volume and dollar value of the individual grease specifications, both synthetic and petroleum base. The composition and equipment application of these greases is presented. A prediction of future procurement of the more common use greases is also made.

THE MILITARY PETROLEUM Supply agency, during the fiscal year 1958, awarded contracts on behalf of the U. S. Military departments, having a total monetary value of \$1,194,007,146.

This figure represents the purchase of petroleum and related products, and certain commercial services in connection with terminalling operations. Table Number 1 illustrates the breakdown of the total procurement dollar for small business and small business setaside versus identical data for the Packaged Products and Specialties branch of the Purchase division, under which branch the procurement of greases is centralized. Of the total procurement dollar value for this branch, \$1,100,178 covers the awards for all types of greases used by the military departments. In pounds of grease, this represents 6,873,430.

A total of twenty-five (25) various types of greases covered by military or federal specifications are assigned to the agency for procurement purposes. Of these, ten (10) are synthetic base greases. All require approval or qualification of a fixed formulation by the government prior to any purchase or procurement. Table Number 2 covers the procurement figures of synthetic base greases for the 1958 fiscal year. The cost

TABLE 1

Dollar Value of Procurement Awarded During Fiscal
Year 1958

	A	gency Totals	Packaged Products & Specialties Branch
Total Procurement (World-Wide)	\$1	,194,007,146	\$36,740,036
Total Procurement (U.S.)	5	842,702,684	\$35,606,412
Total Awarded to Small Business	\$	257,117,751	\$17,413,815
Total Awarded Unde Small Business Set-Aside	\$	49,166,74-	\$ 3,692,730
Percentage of U.S. Total Awarded to Small Business		30.5°	48.9%

of packaging and packing, and transportation to the delivered point where applicabl, are included in the weighted average cost per pound. The actual price per pound, therefore, will vary from the average depending on the container size and transportation charges, if any. The following information is presented concerning those greases where quantity procured or cost per pound may be of interest:

MIL-G-3278 is basically a synthetic low temperature liquid lubricant and lithium soap grease. It is used on ball, roller and needle bearings, gears, and on sliding surfaces in instruments, cameras, electronic gear and aircraft control systems. Volume wise, procurement of this grease should remain fairly constant although a possibility does exist that in the future it may gradually be replaced by MIL-G-7118 grease. Packaging has ranged from eight ounce tubes to 120 pound drums, predominantly in the smaller sizes.

MIL-G-6032 is a synthetic oil and lithium soap grease. It is used on taper plug valves, gaskets, and other applications in fuel and oil systems of aircraft. A stick type may be incorporated in this specification in the future. Past procurement has been in one pound containers.

MIL-G-7118 is a synthetic low temperature oil, lithium soap and extreme pressure additive product. Its application is on aircraft gears, actuator screws and equipment requiring high load carrying capacity. This grease is essentially MIL-G-3278 plus the E.P. additive. As mentioned previously, it may eventually replace MIL-G-3278 grease. It is being procured in one and five pound containers.

MIL-G-7421 in order to meet the low temperature requirement is a synthetic oil and lithium soap grease.

Its temperature range is minus 100°F to plus 225°F and is used on low-torque equipment. Packaging has been in one pound containers.

MIL-G-15793 is a high grade synthetic oil, lithium soap and additive grease. It is used for lightly loaded bearings of fire control equipment and instruments including related components, such as gyros and control mechanism; also for gears, bearings, sliding parts and other small precision instruments and devices. Past procurement has been in one pound containers only.

MIL-G-25537 is a low temperature synthetic liquid lubricant and calcium soap grease. It is used in bearings having oscillating motion of small amplitude and is particularly suited for equipment which must operate at ambient temperatures of minus 65°F to plus 160°F. Being of fairly recent issue there have been no procurements to date, however, its general use in helicopters should result in purchase activity in the future.

MIL-G-25760 is essentially a high melting gelling agent and a wide temperature range liquid lubricant. The composition of the lubricant is not otherwise lim-

TABLE 2 Synthetic Base Greases

Specification			Weighted Average Cost/Pound
MIL-G-3278	Grease; Aircraft & Instruments (For Low and High Temperatures)	212,600	1.0027
MIL-G-6032	Grease, Plug Valve, Gaso line and Oil Resistant	60,000	1.57
MIL-G-7118	Grease, Aircraft Gear & Actuator Screw For Low and High Temperatures	31,600	1.70
MIL-G-7421	Grease; Extreme Low Temperature	5,000	1.74
MIL-G-15793	Grease, Instrument	1,250	2.88
MIL-G-25013	Grease, Ball and Roller Bearing, Extreme High Temperature	None	
MIL-G-25537	Grease, Aircraft; Helicop- ter Oscillating Bearing		
MIL-G-25760	Grease, Aircraft; Ball and		
(USAF)	Roller Bearing, Wide Temperature Range	None	
MIL-L-4343	Lubricating Grease, Pneumatic System	2,100	4.7086
MIL-L-7711	Lubricating Grease (Ger eral Purpose Aircraft)	53,890	0.2161
MIL-L-15719	Lubricating Grease (High Temperature, Electric Motor, Ball and Roller Bearings)	2,010	6.44

ited but substantial proportions of non-petroleum materials will be required to meet the volatility and low temperature requirements of this specification. It is intended for use in ball and roller bearings over the temperature range of minus 65°F to 350°F and is particularly designed for which bearings in internal brake wheel assemblies which often must function at temperatures as low as minus 65°F, yet, for short periods of time during the aircraft braking operation, may have wheel bearing temperatures in excess of 350°F. It may also be suitable for equipment which must operate at both low and high temperatures and which require a grease with good lubricating qualities. Being of recent issue there has been no procurement activity to date, however, gradual use of this grease in lieu of specifi-

remain fairly constant. Procurement has been in one, five, and 35 pound containers.

MIL-L-15719 is a silicone fluid and lithium soap grease. Additives to improve oxidation stability, rust inhibition, mechanical stability, texture and wear resistance may be included. It is a high temperature grease for the lubrication of ball and roller bearings, primarily for class H insulated (silicone) electric motors with heat-stabilized ball bearings. Its normal temperature range is from 0°F to 300°F. It has been purchased only in 8-ounce tubes.

Table Number 3 outlines the fiscal 1958 procurement data of petroleum base greases. Here again the cost of packaging, packing and any transportation to delivered point, is included in the cost figures.

TABLE 3-Petroleum Base Greases

Specification	Product Nomenclature	958 FY Procurement in Pounds	Weighted Average Cost/Pound
MIL-G-2108	Grease, General Purpose No. 2	27,040	.1043
MIL-G-7187	Grease, Graphite, Aircraft Lubricating	5,000	.230
MIL-G-10924	Grease, Automotive and Artillery	5,930,770	.1285
MIL-G-1441	Grease, Railway Brake Cylinder	None	
MIL-G-16908	Grease, Bearing (For General Ordnance Use)	21,600	.1584
MIL-G-17740	Grease, Extreme Pressure	58,500	.183
MIL-G-17857	Grease, Plug Type Scupper Valves	None	
MIL-G-18458 (Ships)	Grease, Wire Rope-Exposed Gear	20,750	.1913
MIL-G-18709	Grease, Ball and Roller Bearing	50,000	.2126
MIL-L-3545	Lubricating Grease; High Temperature	204,755	.245
MIL-L-7645	Lubricating Grease; Lead Soap and Mineral Oil Ba	ase 4,000	.9380
MIL-L-7711	Lubricating Grease (General Purpose Aircraft)	53,890	0.2161
MIL-T-3123	Tail-Packing Compound (For Use in Torpedoes)	None	
VV-G-632	Grease; Lubricating, Automotive and Industrial	179,505	.1115
VV-G-671	Grease, Graphite	3,000	.1525
VV-G-679	Grease, Lubricating, Locomotive	None	

cation MIL-L-3545, should result in future purchase activity.

MIL-L-4343 is a silicone or di-ester lubricant and lithium soap product. It is used as a lubricant between rubber and metal parts of pneumatic systems. It may also be used for pressurized cabin bulk-head grommets and other mechanisms requiring rubber to metal lubrication. No change in the specification requirements nor in volume procured is anticipated. One pound containers have been used for its packaging.

MIL-L-7711 is essentially a non-petroleum oil plus a sodium soap. It is intended for use in anti-friction bearings, gear boxes and plain bearings where reasonably low temperature operation and high temperature stability may be required. Future procurement should

*MIL-L-*2108 is a mineral oil and sodium soap grease. There will be no further procurement of this grease by the Military Departments. In lieu thereof specification MIL-G-10924 grease, automotive and artillery will be utilized.

MIL-G-7187 is a mineral oil, sodium or lithium soap grease with graphite added. It is intended for heavy loaded intermittent applications such as starter gears or slow moving plain bearings and sliding surfaces where graphite will prevent seizure in event of inadequate lubrication. This specification may gradually be replaced by MIL-G-21164 (AER) which is similar to a MIL-G-3278 grease plus molysulfide. Packaging has been in one pound containers.

MIL-G-10924 is primarily a mineral oil and calcium

soap grease. It is used on automotive and artillery equipment under all conditions of service where ambient temperatures of minus 65°F to plus 125°F are encountered. Containers used have been one, five, 35 and 120 pound. The military departments will use this grease in lieu of MIL-G-2108 and VV-O-632 greases. Its future outlook is good, procurement-wise.

MIL-G-16908 is a petroleum oil and lithium soap grease with an anti-corrosion additive and a normal operating range of minus 20°F to plus 150°F. It is intended for the lubrication and corrosion protection of plain ball and roller bearings, and preservation of threads on ammunition. It has been procured in five and 35 pound containers.

MIL-G-17740 is a combination of calcium and lead soaps, extreme pressure additive and a high quality mineral oil. It is used on open or semi-enclosed gears or any sliding or rolling metal surface where load may be high and where the equipment may be exposed to salt spray or moisture. Its operating range may be approximately from 0°F to 140°F. Past packaging has been in five and 35 pound containers.

MIL-G-18458 (ships) is composed of a petroleum oil and such soaps, asphalts, waxes or other additional agents as may be required to conform to the physical characteristics detailed in the specification. It is intended to provide lubrication and corrosion protection for running ropes and exposed gears. Procurement has been in 35 and 120 pound containers.

MIL-G-18709 is a lubricating oil and gelling agent with or without additives. It is used on ball and roller bearings operating at medium speeds and over a temperature range of 125°F to 200°F and for short intermittent service at 225°F. Procurement has been in one, five and 35 pound containers primarily in five pound containers.

MIL-L-3545 is a combination of petroleum oil and sodium or lithium soaps. It is a high temperature lubricating grease intended for the lubrication of aircraft engine accessories but will in many instances, be satisfactory under certain conditions for anti-friction bearings required to start at temperatures as low as minus 40°F. As previously mentioned during the discussion of synthetic greases, this specification will eventually

be replaced by MIL-G-25760. Procurement has been in the one, five and 35 pound containers.

MIL-L-7645 as indicated by the specification title, is a lead soap and mineral oil grease. Other metallic soaps, fatty oils and sulfurized or chlorinated oils may be present. This grease is a semi-solid, non-corrosive, mild extreme pressure lubricant for use in gears, screws, and threads. It has rather limited application and has been procured in one pound containers only.

VV-G-632 as mentioned earlier will not be utilized by the military departments in the future.

VV-G-671 is composed of a well refined mineral oil and calcium soap. It is used in compression grease cups for bearings of machinery operating at temperatures not in excess of 150°F. It has been procured in one, five, and 35 pound containers, primarily in the smaller sizes.

As to future military grease requirements, the outlook is good. Grease application in lieu of oil has gradually increased over the period of years and present indications are that the trend will continue. The extent of increased military procurement is difficult to forecast except for generalizations. Greases intended for application on ground equipment will remain fairly constant with possibly slight yearly increases. A major exception to this will be the procurement of MIL-G-10924 automotive and artillery grease (GAA). A substantial increase over present requirements is anticipated during the next few years as military stock levels of specification MIL-G-2108 general purpose grease and specification VV-G-632 automotive and industrial grease are depleted and replaced by GAA grease. Increases in most greases intended for airborne equipment can also be anticipated. This prognosis is based on the approximately 46 per cent increase in jet fuel requirements through 1961. Due to greater thrust demands, jet engines will be larger and will consume more fuel per engine. However, the larger engines and airframes will require more lubrication points. This in turn, should result in increased grease procurement.

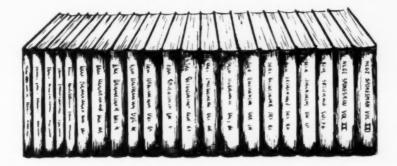
The military departments and the Military Petroleum Supply agency appreciate the cooperation extended by the grease industry in the past and are confident that any problems arising in the future will be resolved in the same spirit.

About the Author

R. J. Horwath attended Purdue university. He has been in the petroleum business for 25 years. For nine years he worked in the research laboratory of the M. W. Kellog company as project supervisor. For eleven years he held the position of field petroleum inspector on the easter seaboard

for the Department of the Air Force. The balance of this period has been with the Armed Services Petroleum Purchasing agency and its successor, the Military Petroleum Supply agency. Mr. Horwath is currently head of the technical data branch, technical division of the latter agency.





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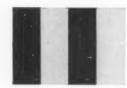
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Literature and Patent Abstracts

Anti-Friction Bearing Lubrication

Ball and Roller Bearing Lubrication

Lubrication, the Texas Co., Vol. 44, No. 8, August, 1958. The nature of grease lubrication and reasons why the majority of rolling-element bearings are now lubricated with grease are discussed. Test techniques are likewise described and a number of graphs and illustrations supplement all of this information. A bibliography lists 32 references, most of which are recent and almost all of which are concerned with application of lubricating greases.

Grease Flow in Shielded Bearings

O'Halloran, Kolfenbach and Le-

land, Lubrication Engineering, p. 104-07, March 1958. By the use of dyed lubricating grease in the bearing and undyed lubricating grease on the shields, it was determined that the lubricant moved as an entity and that there was considerable movement of the lubricant between the bearing proper and the shields. One dye was adsorbed on the soap and another dye was dissolved in the oil of the lubricating grease.

Analysis

Rapid Chromatographic Analysis of Soap-Thickened Lubricating Greases

G. W. Powers, Jr. and F. J. Piehl. Anal. Chem. Jan. 1958, Vol. 30, p. 28-31. This method is appliable to lubricating greases containing petroleum, silicone and ester oils. The precision and accuracy are equal to the Standard ASTM Method. Determinations can be made of four to six samples in eight hours.

Samples of lubricating greases are decomposed with ethane sulfonie acid and then passed through a column containing silica gel. Chloroform is first used to displace the liquid portion of the sample. This is followed by a chloroformacetic acid solution to displace the fatty acids. After evaporating the solvent both fractions are weighed.

Test Methods

Grease Bleeding, Its Mechanisms and Measurement

E. G. Ellis, Scientific Lubrication, 10, No. 9, 20-26 (1958). This article is largely a review and summary of various test methods, both British and American, for determining oil separation in lubricating greases. However, thermal tests which actually measure structure stability are also classified as covering oil separation. Specifically, a calcium base lubricating grease is held in an oven maintained at 120°C. for one hour and then left undisturbed for 24 hours. At the conclusion of the test the sample shall show no signs of oil separation. The British accomplish such stability by the use of some wool grease fatty acids in forming the calcium soap.

The author concludes with the thought that the majority of users of lubricating greases will accept some oil separation during storage of such products.

Separation of Oil from Lubricating Greases in Storage

ASTM Bulletin No. 233, October 1958, pages 27-32. This report to Technical Committee G of ASTM Committee D-2 was submitted by a group of which B. B.



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Farrington was the Chairman.

In a study of mechanism of oil separation it was found:

- a. That differential pressure accelerates oil separation.
- Tension stops or reduces oil separation.
- Temperature increase accelerates oil separation.
- d. Vibration has an accelerating effect on oil separation.
- e. Oil separation is the result of the force of gravity which causes a flow of the oil throughout the affected mass.

A cratered pail test was found reliable for evaluating the tendency of greases to separate oil in storage. In the test three craters are made in a 35-lb. test pail. Cones 3 in. in diameter and 4 in. deep, fabricated from 8-mesh, 0.028 in. diameter wire screen, are inserted into the grease and the grease then removed from the cone. Oil separation is determined by calculating the average volume of oil in the crater from the surface diameter of the pool of separated oil. A total time of four weeks was used for observation.

The conclusions resulting from this observation were:

- a. During accelerated laboratory tests, pressure and temperature should not deviate markedly from those encountered in bulk storage. Pressure of 1 psi and temperature of 130°F should not be exceeded.
- b. A 3-oz. crater test and also Federal test method 322-T correlate well with the cratered pail test in the case of three types of greases, namely two sodium-calcium base products with oils of 166.8 and 223 viscosity SSU at 100°F and a lithium base product with an oil of 66.8 viscosity SSU at 100°F.
- c. The suggested method, namely cratered pail test, evaluates the tendency of lubricating greases to separate oil in storage.

Compositions

Lubricating Greases Thickened with Polyamido Acid Salts

Hotten (U. S. Patent 2,849,400 assigned to California Research corporation) states that if metal salts of polyamido acids are used as thickeners for lubricating oils the resulting products are oxidation and water resistant. Also it is claimed that such bodying agents do not lose their thickening action at increased temperatures as rapidly as do metal soaps of fatty acids.

The polyamido acids are formed in a two stage reaction. First, a monobasic acid containing 10 to 22 carbon atoms is reacted with an alkylene polyamine, such as diethylene triamine, triethylene tetramine, propylene diamine, tetrapropylene pentamine, etc., to form a carboxyamide. One mol of this carboxyamide is then reacted with one mol of a dibasic acid, such as oxalic, adipic, isophthralic, etc., to form polyamido acid.

An example of manufacture indicates the preferred compounds and the method of preparation. A mixture of 284 grams of stearic acid and 206 grams of diethylene triamine was heated with agitation at 390°F. to remove water formed. To the cooled mixture 500 grams of adipic acid was added and the mass was heated at 390°F. for about 2 hours to remove the water of reaction.

A mixture of 30 grams of the above acid and 170 grams of a California solvent-refined naphthenic base oil having a viscosity of 450 SSU at 100°F. was heated at 460°F. until solution was complete. After cooling 3.7 grams of sodium hydroxide in 10 ml. of water was added, followed by again heating to 460°F., cooling, and finally milling through an 80 mesh screen.

The resulting lubricating grease had an ASTM dropping point of 336°. A five-gram ball of the lubricant immersed in boiling distilled water was intact after 60 minutes.

In U. S. 2,849,401 Hotten describes the use of diamido acid salt

thickeners for lubricating greases. Thus, one mol. of stearic acid was reacted with one mol. of ethylene diamine followed by a further reaction with one mol. of adipic acid. Thirty grams of the resulting diamido acid was mixed with 170 grams of oil and then converted to a lithium salt.

When the above mixture was heated to 500°F., pan cooled and milled by screening, a lubricating grease resulted which had an ASTM penetration of 278 and a dropping point of 376°F. In a boiling water test the product was intact after 120 minutes.

An aluminum compound of a similar diamido acid was found to produce a smooth lubricating grease. Likewise a calcium salt was used as a thickener for oil to give a lubricant with a dropping point of



191°F. When lauric acid was reacted with ethylene diamine before heating with adipic acid a diamido acid was formed which gave a sodium salt with a higher melting point than when stearic acid was used. The resulting lubricating grease had a dropping point of over 500°F.

E. P. and Gear Lubricants

The Extreme-Pressure Lubricating Properties of Some Sulphides and Disulphides, in Mineral Oil, as Assessed by the Four-Ball Machine

Davey and Edwards in a new publication 'WEAR', Vol. 1, p. 291-304, February, 1958, conclude from the above investigation that blends of disulphides in mineral oil show superior extreme-pressure properties to blends of monosulphides containing the same amount of total sulfur. For a given load, wear was higher for a disulphide blend than for one containing the same proportion of free sulfur. The mechanism of the action of these compounds is discussed and indi-

rect evidence is obtained for the formation of ferrous sulphide films on steel by disulphide blends under extreme-pressure conditions.

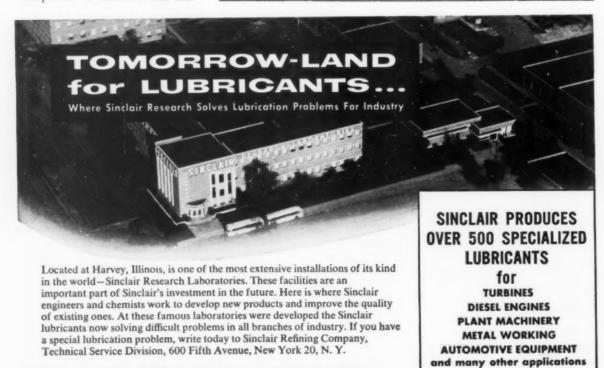
Extreme Pressure Lubricants

Compositions which satisfy both low torque-high speed and high torque-low speed operating conditions in gear sets consist or a mineral oil containing eight to twelve weight per cent of a dipentenetreated reaction product of a mineral lubricating oil and phosphorus pentasulfide, formed at a temperature of 200 to 600°F., and 0.5 to 2 per cent of chlorodibenzyldisulfide.

The benefit of such a combination is indicated in the tabulation below. Here additive A consists of a phosphosulfurized hydrocarbon and additive B is chlorodibenzyl disulfide. The base oil stock was an SAE 90 in each case.

These E.P. lubricants are described in U.S. Patent 2,827,433, issued to Fischl, Waddey and Beyer-

		TABLE 1		
Oil	Additive Used	Per cent Chlorine	Full-Scale L-19	Gear Test L-20
I	None	Nil	Fail	Fail
1	10% A	Nil	Fail	Pass
I	9% A, 1% B	0.13	Pass	Pass
H	None	Nil	Fail	Fail
11	9% A, 1% B	0.13	Pass	Pass
III	None	Nil ·	Fail	Fail
111	10% A	Nil	Fail	Pass
III	9% A, 1% B	0.13	Pass	Pass



stedt and assigned to Esso Research and Engineering company.

Gear Oil Compositions

Gear oils suitable for use in automotive vehicles and industrial machines are improved by the addition of an ester-type oiliness agent according to Manteuffel, Brennan and Stucker in U. S. Patent 2,830,-952, assigned to The Pure Oil Co. Specifically, the E.P. lubricant consists of mineral oil to which has been added eight to fifteen per cent of a sulfurized and phosphorized lard oil, sperm oil or cotton-seed oil and 1 to 21/2 per cent of an alkyl ester, such as methyl ricinoleate. For example, when tested on a Four-Ball machine a composition containing twelve per cent of a treated lard oil gave a mean Hertz load of 37 kg. After addition of 21/2 per cent of methyl ricinolcate the value was increased to 54.9 kg.

ASLE Transactions

The above publication will be issued semi-annually and will contain papers not published in *Lubrication Engineering*. Volume 1, No. 1 contains two articles of interest to Spokesman readers, abstracts of which follow.

Grease Lubrication of Ultra-High Speed Rolling Contact Bearings

By J. B. Accinelli, page 10. Tests were made of 20 and 25 mm. ball bearings at speeds up to 100,000 rpm. Mechanical factors were found to have more influence on performance than lubricating grease variables. Best results were obtained with such lubricants of smooth tecture and hard consistency, that is No. 3 or No. 4 NLGI Grades.

In one series of tests lubricating greases containing four different thickeners were used, that is fine silica, clay, lithium hydroxy stearate and sodium beeswax soap. The "rst three, which were smooth textured, gave approximately the same temperature time histories irrespective of the oil viscosity. On the other hand, the sodium beeswax soap product gave very high temperature rise during a much longer breakaway period and leveled off

at a temperature above that of the other lubricating greases.

The difficulty of maintaining adequate amounts of lubricant against the action of centrifugal force in the critical areas explains in part the poor lubricating experience with greases. It was thought that high bleeding might be an advantage in such products.

One run of 106 hours to failure was made with a MIL-G-3278 grease finished to a No. 4 grade but several attempts to duplicate this run were unsuccessful.

Dynamic Oxidation Test to Evaluate Grease Performance at High Temperatures

Henry E. Mahncke and David J. Boes. p. 17. A bearing was operated in an oxygen atmosphere and the rate of oxygen consumption measured to indicate grease failure. By this means the mechanism of grease failure over a temperature range of 70 to 250°C. can be studied. Two types of silicone greases, not other-

wise identified, were tested at 200°.

While the apparatus was designed to study lubricating grease and its behavior at high temperatures, after a few test runs it became evident that the bearings themselves were responsible for most failures. Although tests were run on the same quantity of lubricant under the same conditions of temperature and atmosphere, life spans of the lubricating greases varied from 1.5 to 240 hours.

The smaller the test bearing's initial clearance the shorter the grease life and the poorer the test reproducibility. Thus, with internal clearances of -0.0001 to +0.0001 inch, failures occurred after five to ten hours operation. On the other hand, when clearances were 0.0008 to 0.0013 inch good reproducibility and lower running temperatures occurred.

A factor affecting test reproducibility and grease life was explained as follows. As the lubricant is oxi-





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dized, varnish and gums are formed which accumulate on the bearing components, such as the race shoulders, the balls, and even the sides of the ball race itself. Eventually a piece of dried material falls into the ball path. The lack of sufficient clearance between the balls and the races prevents the balls from wiping this obstruction out of their path. This results in a subsequent rise in the operating temperature of the bearing and therefore further and more rapid oxidation, which in turn causes increased running torque, an additional rise in temperature and further accumulation of oxidized lubricating grease.

Miscellaneous

Lubricating Greases Improved as to Oxidation and Worker Stability

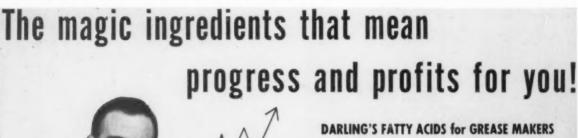
According to British patent 794, 619, addition of 0.1 to 5 per cent of a stabilizing agent improves the oxidation resistance and worked sta-

bility of lubricating greases consisting of lubricating oil thickened with alkali or alkaline earth metal soaps. The stabilizing agent is the reaction product of a phenolic-amino and ethylene oxide. The resin is obtained by condensing an alkylphenol with formaldehyde, or a polymer of the same, in the presence of ammonia. For example, a mixture of 100 parts of p-iso-octyl phenol, 50 parts of mineral oil and 20 parts of trioxymethylene was heated for six hours to 130 to 150° F. while fifteen parts of ammonia were injected. This was followed by heating the mixture for two hours at 212 to 220°F, and finally for three hours at 300°F, to give a resin containing 2.2 per cent of ni-

The stabilizing agent was then obtained by reacting this resin with 25 per cent by weight of ethylene oxide at 320°F for 20 hours.

A lubricating grease was made

from 15 per cent stearic acid, 2.45 per cent sodium hydroxide and 82.55 per cent of a mineral oil having a viscosity at 100°F. of 32 cs. and a V. I. of 115. This product, after being pan cooled, had the following characteristics: drop point °F. 173 (while the patent so states it would seem more logical that this figure is in °C.); flow test when 100 g. was held on a 100 mesh wire gauze cone for 50 hours 9 per cent; ASTM penetrations 50 strokes 350, 1000 strokes 370, 100,000 strokes fluid; bomb oxidation test, 10 hours to obtain a pressure drop of 0.35 kg/cm² at 99°C. After 1.5 per cent of the stabilizing agent was incorporated in this lubricating grease the following characteristics were noted: drop point °F. 183, flow test 0.8 per cent; ASTM penetrations, 50 strokes 283, 1000 strokes 308, 100,000 strokes 325; bomb oxidation test 600 hours to obtain a pressure drop of 0.35 kg/cm² at 99°C.





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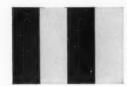
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People in the Industry

B. G. Symon Made Vice-President of International Lubricant Corp.

Benjamin G. Symon, formerly industrial products manager of Shell Oil company, has been named vice president of International Lubricant corporation of New Orleans. International manufactures and compounds greases and lubricating oils.

Symon has been a director of the corporation for several years. He was recently made general sales manager.

Symon, a mechanical engineer-

ing graduate from the University of Missouri, joined Shell in 1928 as assistant to the manager of the lubricants department in St. Louis. In 1940 he moved to New York as manager of the technical products department and was named manager of the lubricants department two years later. He was named manager of Shell's industrial products department in 1955.

Symon is an active member of the American Petroleum Institute, a director of the National Petroleum association, a member of the American Society of Lubricating Engineers, and a past president (1949) and former director of NLGI.

Nopco Appoints M. Ernest Ionescu

Robert F. McClellan, vice president of Nopco Chemical company, announced the appointment of M. Ernest Ionescu as manager of the newly-formed special products department of the industrial sales division, effective November 1.

The new department has been established to develop markets for the anti-oxidant, BHT (butylated hydroxy toluene), used extensively in the petroleum industry.

Mr. Ionescu was educated in Austria and Germany, and at Rensselaer Polytechnic Institute.

Mullin Is Sales Manager

The appointment of Gerald W. Mullin as sales manager-lubricating equipment for Southern California, was announced by C. A. Stutzman, West Coast manager of this division of Aro Equipment of California, with headquarters in Los Angeles.

Mr. Mullin has acquired many years' experience in handling petroleum marketing equipment and is already thoroughly familiar with Aro lubricating equipment for automotive, industrial and farm fields. For the past two years he has been associated with A. R. Sedgebeer, Southern California division manager.

The main plant and general offices of Aro are in Bryan, Ohio.

McCumber Is Midwest Representative

H. A. Mayor, Jr., executive vice president of Southwest Grease & Oil Co., Inc., recently announced the appointment of Mr. G. L. "Mac" McCumber as the com-

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Acid value	197—203	195—201
Saponification value	198—205	197—203
Unsaponifiable content	1.5% max.	2.0% max.
Polyunsaturates	3% max.	1

We would like you to see our Oleic Acids and compare them critically with other competitive products, so you may fully appreciate Century Brand quality. We invite your comparison of Century Brand Oleic Acids because only you can realize their advantages in *your* products.

A request to Dept. H-30 for samples will receive prompt attention and we will welcome the opportunity to put these better products in your hands.



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pany's midwestern sales representative. Mr. McCumber will replace Jack Hodges, who has been promoted to coordinator of sales and service at the home office in Wichita, Kansas.

McCumber joined Southwest in September and has been participating in the company training program. The Southwest training further supplements his fourteen years' experience in the industry. "Mac" has been fortunate in obtaining several years' experience in the sales department of the Gulf Oil corporation, in addition to a number of years as division sales promotion manager and retail specialist with the D-X Sunray Oil company.

Centracco Is Manager

V. J. Centracco has been appointed by Kerr-McGee Oil Industries, Inc., as manager of the Chicago area industrial sales department of the general sales division. Centracco formerly was employed by Anderson-Prichard as a manager in their St. Louis office.

Wallace & Tiernan Elect

Wallace & Tiernan, Inc., Belleville, N. J., has announced the election of Robert M. Jackson as president and T. T. Quigley and C. H. Rybolt as vice presidents: H. A. Gilman was named treasurer. Wallace & Tiernan, Inc., are manufacturers of organic peroxides, fatty acids and other products.

SERVICE AIDS

Send Orders to: National Lubricating Grease Institute, 4638 Nichols Parkway, Kansas City, Mo.

BONER'S BOOK—Manufacture and Application of Lubricating Greases, by C. J. Boner. This giant, 982-page book with 23 chapters dealing with every phase of lubricating greases is a must for everyone who uses, manufactures or sells grease lubricants. A great deal of practical value. \$18.50, prepaid.

NLGI SPOKESMAN



Industry News

Sonneborn Distillation Unit Nears Completion

A new distillation unit is nearing completion at the L. Sonneborn Sons, Inc., Franklin, Pennsylvania, refinery, according to Mr. L. C. Borell, refinery manager. Several weeks ago, the unit's 80-foot tower, weighing 30 tons, was erected by an outside contractor and, since then, the refinery's own construction crew has been handling the large amount of pipe-fitting, insulation, and steelwork required to prepare the unit to go on-stream.

Primary purpose of the new distillation set-up is to increase the refinery's capacity for turning out specialty solvents, which are manufactured as important by-products of a growing lube oil operation. The principal products of the Sonneborn refinery at Franklin are Amalie lube oils and greases. The main raw material for these products is Pennsylvania grade crude oil. Among these by-products are petrolatum grease, microcrystalline wax, and specialty solvents.

The Sonneborn organization is an important factor in the petroleum products field. Its Amalie lubricants have been sold for fifty years under this particular brand name. It is a leader in the field of petroleum-based pharmaceuticals such as white oil and petroleum. Its petroleum sulfonates find wide use as additives both in its own and other refiners' lube oils.

Sinclair to Divide Marketing Territory

Sinclair Refining company announced division of its thirty-six state marketing territory into two regional territories. L. W. Leath, vice president and general sales manager, has been placed in charge

of the Western region, and L. J. Hoar, vice president and newly appointed general sales manager, in charge of its Eastern region.

Spokesman for the company said these organizational changes have been made to achieve greater coordination and concentration of sales efforts in the widespread territory in which Sinclair operates.

Votator Bulletin Describes Lubricating Oil Packaging

Use of the high-speed Votator filler for continuous, automatic product flow in packaging lubricating oils, antifreeze and related products is described in a new brochure (PED-259) available from Girdler Process equipment division of Chemetron corporation, Louisville, Ky.

Operating speeds as high as ten one-quart containers per second are among the specifications listed for the unit, the rate varying with product viscosity, fill temperature and container design.

Applications mentioned for the piston filler include packaging of lubricating oil, upper cylinder lubricant, friction-proo ng lubricant, brake fluid, antifreeze, radiator-flush solvent and various other liquids including sprays, deodorizers, waxes and cleaners.

American Can Company Research Spurs New Era of Can Manufacture

The motor oil can appears to be on the threshold of important advances. Introduced by American Can company in 1932, this can already has been largely responsible for revolutionizing the marketing of motor oil.

The petroleum industry was

among the first to recognize the individually labeled, tamper-proof metal can as a much needed package compared with the crackerbarrel era of dispensing oil from bulk containers. This initiative by the petroleum leaders led to can makers providing a package that would offer consumers a product of indisputable standardization and quality.

Success of the metal motor oil can is recorded in the production statistics of the can-making industry. In 1947, U. S. Motorists used up almost 1.2 billion cans of motor oil, American Can company reports. By 1956, that figure had

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Harshaw Lead Base, as an additive to petroleum lubricants, improves extreme pressure characteristics and imparts the following desirable properties:

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Improved wetting of metal surfaces
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1945 E. 97th Street • Cleveland 6, Ohio Branches In Principal Cities advanced to about 1.8 billion, and it is estimated that production will reach around two billion cans in 1958, representing well over 80 per cent of all the motor oil marketed in this country.



CRYSTAL ball is really a high-vacuum evaporator.

Other types of containers for lubricating grease products also have made large gains during recent years. Canco's new "Accu-Por" container with its non-drip nozzle has become popular for oil for outboard motors, lawn mowers, small generators, chain saws and other automotive and marine uses. Oblong cans, pressure containers and collapsible metal squeeze tubes have found large markets for a multitude of lubricants and greases.

Behind this packaging phenomenon lies a story of vast research and production skills—a story that is still going on, with even greater possibilities for the future.

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The House of Good Grease needs an aggressive and ambitious man 25 to 35 years of age to train for a company sales representative. The position requires extensive travel and may mean relocation. We desire previous lubrication sales background and some college academic work in Engineering, Chemistry or Geology. For details, call or write:

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Several years ago a change to a tin-free all-steel container was made possible by two Canco developments. The first was a special highbaked oleoresinous enamel coating to replace the thin tin plating on steel sheets from which the cans were made. The second was the development of a thermoplastic cement to replace the traditional solder composed of an alloy of tin and lead-both strategic materials -which seals the container sideseams together. This latter feature permitted around-the-can lithography, thus providing the modern motor oil can with additional protection against corrosion and oxidation. For further protection an aluminum coating was added to the can ends, making the entire outside surface of the container resistant to atmospheric or storage conditions ordinarily detrimental to steel.



A CANCO researcher uses an ultramodern resin reaction kettle to synthesize experimental high-polymer canlining materials.

Can research is, however, not stopping here. William C. Stolk, president of American Can, said "in the can-making revolution that lies before us research will play a major role in obsoleting present-day methods and bringing about techniques unrecognizable by today's standards."

Mr. Stolk pointed out that not only aluminum is being investigated by Canco's Barrington, Illinois research center-largest in the industry-but that long-range research is making real progress with many new materials.

"We are concerned primarily to make cans from any material that will provide us with a can that is as good or better than the ones we have today," Mr. Stolk said. "Our research is concerned with many different metals or metal combinations, also plastics, paperboard, laminates, foils and other materials."

Coil Processing Program

Another major step in the industry's can-making "revolution" has been taken by American Can company through its \$31 million coil processing program, part of a longrange technological development designed to produce containers of ever better quality and at the lowest possible cost. The company has installed a nation-wide network of coil cutting installations where huge coils of tin plate and steel plate, weighing up to 16,000 pounds, are sheared into sheets for making cans -a process traditionally performed by steel mills.

Canco's research is also advancing to a point where it may be possible to incorporate enameling and decorating units in the coil lines. The ultimate in this technological evolution would be to run the inspected, treated and enameled directly off the coils into the canmaking machine, completely bypassing the sheeting operation.

Canco points out that in the history of can manufacturing every major development has materially

Continued on page 488

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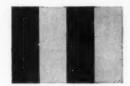
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Future Meetings

FEBRUARY, 1959

- 1-6 ASTM Committee D-2 meeting, Sheraton-Jefferson Hotel, St. Louis.
- 16-17 Packaging Institute, Petroleum Packaging Committee, Goodhue Hotel, Port Arthur, Texas.
- 26-27 API Division of Marketing, Lubrication Committee Meeting.

*MARCH, 1959

3-5 SAE Passenger Car, Body, and Materials Meeting, Sheraton-Cadillac, Detroit, Mich.

APRIL, 1959

- 5-10 American Chemical Society 135th National Meeting.
- 15-17 National Petroleum Association, Semiannual Meeting. Hotel Cleveland, Cleveland, Ohio.
- 21-23 ASLE Annual Meeting and Exhibit, Hotel Statler, Buffalo, New York.

MAY, 1959

- 4-6 API Division of Marketing, Lubrication Committee Meeting. San Marcos Hotel, Chandler, Ariz.
- 15-24 International Petroleum Exposition
- 27-29 API Division of Marketing, Midyear Meeting. The Savoy, Des Moines.
- 31-June 6 Fifth World Petroleum Congress. The Coliseum, New York City.

* Tentative

JUNE, 1959

- 14-19 SAE Summer Meeting, Chalfonte-Haddon Hall, Atlantic City, N. J.
- 21-26 ASTM Annual Meeting Chalfonte-Hadden Hall, Atlantic City, N. J.

SEPTEMBER, 1959

19-21 ASLE and ASME Joint Lubrication Conference, Sheraton-McAlpine Hotel, New York City.

OCTOBER, 1959

- 26-28 NLGI Annual Meeting, Roosevelt Hotel, New Orleans, La.
- 11-15 ASTM Committee D-2 Meeting.

APRIL, 1960

19-21 ASLE Annual Meeting and Exhibit, Netherland-Hilton Hotel, Cincinnati, Ohio.



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Peotone, Illinois Representative-S. A. Bennett

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Cleveland Container Company

4925 So. Halsted St., Chicago 9, Ill. Representative—R. D. Sayles

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6700 South LeClaire Ave., Chicago 38, Ill. Representative-Henry Rudy

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Republic Steel Corporation

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Rheem Manufacturing Company

400 Park Ave., New York 22, New York Representative—F. J. Blume

Rieke Metal Products Corporation

Auburn, Indiana Representative—Glenn T. Rieke

Steel Package Division of **National Lead Company**

722 Chestnut Street, St. Louis 1, Missouri Representative—Warren T. Trask

United States Steel Products

Division, United States Steel Corporation 30 Rockefeller Plaza, New York 20, N.Y. Representative-Wm. I. Hanrahan

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3249 East 80th St., Cleveland, Ohio Representative—Lee Witzenburg

Gray Company, Inc.

60 Northeast 11th Ave., Minneapolis 13, Minn. Representative—B. A. Beaver

Lincoln Engineering Company

5701 Natural Bridge Ave., St. Louis 20, Mo. Representative-G. A. Hubbard

Stewart-Warner Corporation

Alemite Division 1826 Diversey Parkway, Chicago 14, Illinois Representative-E. G. Wicklatz

Trabon Engineering Corp.

28815 Aurora Rd., Solon, Ohio Representative—E. W. Baumgardner

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Derby Refining Co.

420 West Douglas, Wichita, Kansas Representative—W. B. Neil

D-X Sunray Oil Company

Mid-Continent Bldg., P. O. Box 381, Tulsa, Okla. Representative—J. W. Basore

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100 East Ohio Street, Chicago, Illinois Representative—S. F. Graham

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Association, Inc.

245 North High Street, Columbus 16, Ohio Representative-Walter N. Callahan

Valvoline Oil Company

Division of Ashland Oil & Refining Co. Box G, Freedom, Pennsylvania Representative D. A. Smith

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Girdler Process Equipment Division, Chemetron Corp.

P. O. Box 43, Louisville 1, Kentucky. Representative-J. E. Slaughter, Jr.

Manton-Gaulin Mfg. Co., Inc.

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American Potash & Chemical Corp. E. I. du Pont de Nemours & Co.

99 Park Avenue, New York 16, N. Y. Representative—W. F. O'Brien

Archer-Daniels-Midland Company

Chemical Products Division P. O. Box 532, Minneapolis 40, Minn. Representative-J. H. Kane

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Darling & Company

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Wilmington, Delaware Representative-R. O. Bender

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Wallace & Tiernan, Inc.

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Representative-W. J. O'Connell

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The Lubrizol Corporation

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Mallinckrodt Chemical Works

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The McGean Chemical Co.

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Metasap Chemical Company

A Subsidiary of Nopco Chemical Co. 60 Park Place, Newark, New Jersey Representative-T. J. Campbell

Monsanto Chemical Company

800 North Twelfth Blvd., St. Louis 1, Mo. Representative-I. W. Newcombe

Newridge Chemical Company

7025 West 66th Place, Chicago 38, Illinois Representative—T. E. Shine

M. W. Parsons—Plymouth, Inc. 59 Beekman St., New York City 38, New York Representative—Herbert Bye

Synthetic Products Company 1636 Wayside Rd., Cleveland 12, Ohio Representative—Garry B. Curtiss

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Vegetable Oil Products Co., Inc.

Vopcolene Division 5568 East 61st Street, Los Angeles 22, Calif. Representative—C. F. Williams

Witco Chemical Company

122 East 42nd St., New York 17, New York Representative—E. F. Wagner

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1 Market St., West Warwick, Rhode Island Representative—Alberic T. DiMasi

Veresit-Fabrica de Productos Quimicos S.R.L.

Monasterio 271, Buenos Aires, Argentina Representative-Dr. Alexander Erdely

Continued from page 484

benefitted the canner and packer. either directly or indirectly. Better cans have resulted, with increased shelf life or product quality. Taking the plain unlitho-graphed No. 2 can as an example, a half-century ago these containers cost \$14.00 per thousand. Now they average about \$35.00 a thousand. In 50 years the cost of the No. 2 can has gone up only two and one-half times. However, the company states, had the industry not steadily progressed from 1908 methods, the cost of their manufacture would be at least eleven times greater than it is-costing around 15 cents, rather than the actual price of about three and one-half cents.

Through broadened research, investment and continuing technological advances, the can company envisions a new era of container manufacture that would have been thought impossible only a few years ago.

Girdler Catalysts Opens N. Y. Office

Establishment of a New York field office for Girdler Catalysts, Louisville, Ky., manufacturer of specialty catalysts for chemical, petroleum and gas processes, is announced by the chemical products division of Chemetron corporation.

James V. Weir, an experienced administrator and research man in the fields of petroleum cracking and specialty catalysis, will head the new office, the company said. It will provide technical sales and services

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Petroleum and Chemical Engineers

307 E. 63rd STREET KANSAS CITY 13, MO.

"In Engineering it's the People that count" to users of Girdler catalysts in twelve northeastern states including New England, the Middle Atlantic states, Delaware, Maryland and Virginia.

The New York office-at 79 Wall street-is the second of its kind for Girdler Catalysts, following by a few weeks the establishment of a Houston office to serve Texas, Oklahoma, Louisiana, Arkansas and Mississippi. Purpose of the field locations, according to Girdler Catalysts' general manager Howard D. Hartough, is not only closer customer contact, but also to provide technical services relating to application of existing catalysts to new processes, use of new catalysts being introduced by the company, plant design or modification and improvement of processes in which specialty catalysts are required.

Longer Auto Life, Lower Repair Bills Promised

Front end shimmy, vibration, and annoying squeaks and noises, caused by wear on steering assemblies and front end suspension—familiar problems which have plagued motorists for years, may now be significantly reduced with Molysulfide chassis greases.

According to E. E. Smith, manager, chemical sales, Climax Molybdenum Co., a division of American Metal Climax, Inc., new chassis greases containing Molysulfide soon to be available nationally, promise considerable relief from these irritating, sometimes costly problems.

Smith reported that a two-year, two-million-mile research program just completed by a large research institute in the Southwest proves significantly reduced wear in steering assemblies (38 per cent) and suspension points (26 per cent) for automobiles. This means that the motorist can now look forward to easier steering, safer more comfortable driving and reduced repair bills when using "Moly" grease, he claims.

This unusual research program the most extensive testing of chassis greases ever conducted—involved exhaustive tests on fleets of automobiles, trucks, and buses through two years and two million miles of actual in-use commercial service.

Commemorative Stamp for Oil Industry

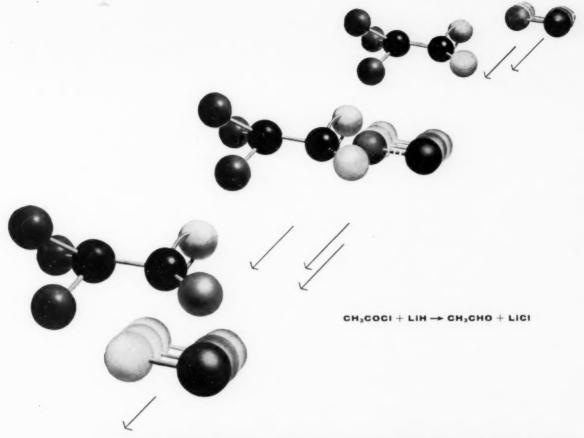
A special commemorative stamp will be issued in 1959 to mark the centennial of the U. S. oil industry, the American Petroleum Institute was advised today by the Post Office department.

The stamp will be issued at Titusville, Pa., on August 27, 1959—"birthplace" of the American oil industry, and 100 years to the day that the first commercial oil well became a reality.

API President Frank M. Porter said the denomination, size, and color of the petroleum stamp will be announced later by Postmaster General Arthur Summerfield.

He expressed gratification, on behalf of the industry, at the selection of oil's centennial for the special recognition.





making the most of lithium hydride

Lithium hydride is one of the most versatile metal hydrides available to the chemist. Long known for its unique solubility properties as compared with other alkali metal hydrides, this lithium compound has amassed an impressive number of applications—both present and future—that go far beyond original expectations. A few examples are in order:

. . . as a reducing and condensing agent

Lithium hydride can convert carbon dioxide to free carbon...can reduce acetyl chloride to acetaldehyde and lithium chloride (illustrated above)...can be used to prepare new hydrides which would otherwise be unobtainable except in small yields and by difficult synthesis...functions efficiently in many organic condensation and reduction reactions...and can easily be increased in solubility or controlled in reactivity by conversion to mixed hydrides.

. . . as a catalyst

Lithium hydride reacts with alcohols to form lithium alcoholates and hydrogen. This reaction makes possible the convenient preparation of anhydrous lithium alcoholate which is useful as an alcoholysis catalyst.

... as a hydrogen bank

Lithium hydride is an ideal source of hydrogen... just one pound of lithium hydride will generate as much as 45 cubic feet of hydrogen gas at S.T.P. This gives you more hydrogen per unit of weight than can be secured by using "bottled" gas in steel containers.

. . . as a nuclear shielding material

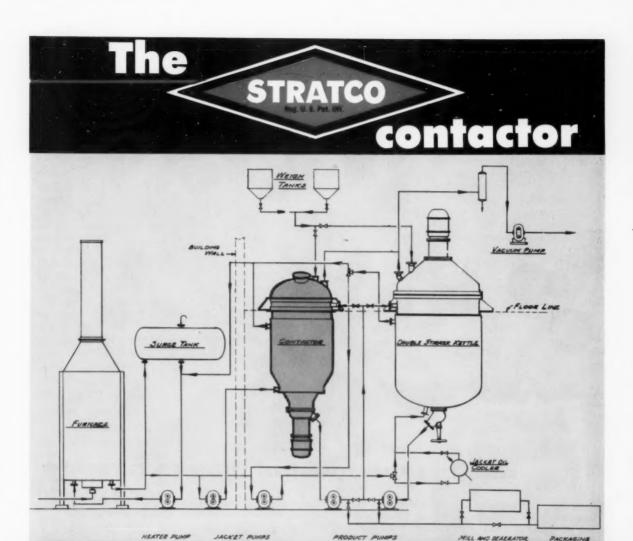
 $N_{\rm H}$ of lithium hydride is 5.90 compared to 6.68 for water at room temperature. And because of its low dissociation pressure at its melting point (27 mm at 680°C.), lithium hydride can be heated to red heat in a thin-wall container . . . without requiring a pressure shell. It appears to be stable indefinitely at this temperature.

These and many other useful characteristics of lithium hydride may help improve your product or process. For complete technical data, write for Bulletin 102. Address request to Technical Literature Dept., Foote Mineral Co., 402 Eighteen West Chelten Bldg., Phila., 44, Pa.



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and How It Simplifies Grease Making

The Stratco contactor is a highly efficient mixing device and when combined with the heating and cooling system shown above provides extremely close control of reaction temperature. With intimate contact between reactants and controlled temperature, very short batch time cycles are required.

Compared with other systems, Stratco

grease plants produce more uniform greases with less soap and require less laboratory control.

A complete Stratco plant layout is illustrated above. Equipment is adaptable to modernization programs as well as new installations. Specific equipment recommendations made without obligation.

STRATFORD ENGINEERING

612 West 47th St.

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PETROLEUM REFINING ENGINEERS

Kansas City 12, Mo.